(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 245 262 A1

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
02.10.2002 Bulletin 2002/40

(51) Int Cl.7: **B01D 39/20**, B01D 53/94, B01J 35/10, F01N 3/28, B01J 35/04, C04B 38/06

(21) Application number: **02007022.3**

(22) Date of filing: **27.03.2002**

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE TR
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: **28.03.2001 JP 2001093412**
08.02.2002 JP 2002032780

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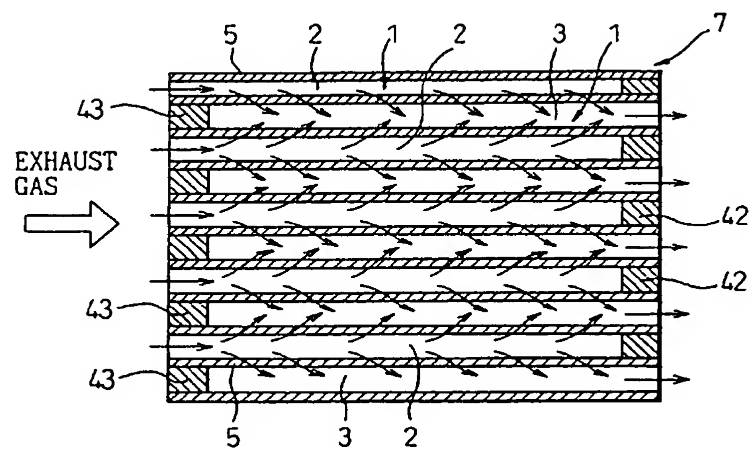
(54) **Exhaust gas purifying filter and method of manufacturing the same**

(57) An exhaust gas purifying filter that does not break and melt and has high capacity to remove particulate matter, and a method of manufacturing the same, are provided.

The exhaust gas purifying filter 7 is made of a ceramic material in a honeycomb structure comprising introduction passages 2 for introducing exhaust gas that includes particulate matter emitted from an internal combustion engine, porous walls 5 that collect the particulate matter and exhaust passages 3 for exhausting the exhaust gas after the particulate matter has been removed therefrom, with the porous walls supporting a catalyst for oxidizing and removing the particulate matter, wherein the porosity of the porous wall is in a range from 55 to 80%, the mean pore size is in a range from 30 to 50 μm, and the total volume X of the pores included in the exhaust gas purifying filter and the volume Y of the pores that are not smaller than 100 μm satisfy the relation of inequality $Y/X \leqq 0.05$.

Fig.2



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porous walls supporting a catalyst for oxidizing and removing the particulate matter, the porosity of the porous walls being in a range from 55 to 80%, and mean pore size being in a range from 30 to 50 μm, while the total volume X of the pores included in the exhaust gas purifying filter and the volume Y of the pores that are not smaller than 100 μm satisfy the relation of inequality $Y/X \leqq 0.05$, wherein the exhaust gas purifying filter is made by firing a preform made in honeycomb structure from a mixture of ceramic powder and a foaming material with the foaming material expanding during the firing process, and adding the catalyst to be supported thereon.

[0013] According to the second aspect of the invention, the pores can be formed with the porosity and the mean pore size described above, as the foaming material expands when dried.

[0014] While the prior art technology is required to use a ceramic powder of large particle sizes in order to form relatively large pores having mean pore size of 40 μm or larger, ceramic powder of large particle sizes makes it difficult to form the exhaust gas purifying filter by extrusion molding. According to the second aspect of the invention, since the foaming material that expands when dried is used, it is not necessary to use a ceramic powder of large particle sizes and the exhaust gas purifying filter of honeycomb structure that has the mean pore size described above can be manufactured by the extrusion molding process.

[0015] Thus the second aspect of the invention makes it possible to manufacture the exhaust gas purifying filter of the first aspect of the invention that has the high performance described above.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016]

Fig. 1 is a front view of the exhaust gas purifying filter of an embodiment of the invention.
Fig. 2 is a sectional view of the embodiment taken along lines II-II in Fig. 1.
Fig. 3 shows pores in the exhaust gas purifying filter and particulate matter passing through the pores according to the embodiment.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0017] In case the porosity of the porous wall is less than 55% in the first aspect of the invention, pressure loss becomes too high for practical use due to buildup of the particulate matter on the surfaces of the porous walls. When the porosity is higher than 80%, strength of the exhaust gas purifying filter may become too low.

[0018] When the mean pore size of the porous walls is smaller than 30 μm, the particulate matter accumulates on the surface of the porous walls and hardly enters the inside of the porous walls. When the mean pore size is larger than 50 μm, the particulate passes through the porous walls because the pores are too large, resulting in a lower particle capturing efficiency.

[0019] In the first aspect of the invention, the total volume X of the pores included in the exhaust gas purifying filter and the volume Y of the pores that are not smaller than 100 μm satisfy the relation of inequality $Y/X \leqq 0.05$.

[0020] As will be described later and shown in Fig. 3, as the particulate matter has sizes in a range from 0.1 to 20 μm, the particulate matter has a high probability of passing through the pores that measure 100 μm or more across. Thus in the case of $Y/X > 0.05$, the efficiency of capturing the particulate matter may decrease. It is more preferable that all the pores are smaller than 100 μm.

[0021] The catalyst is preferably a noble metal catalyst, since a noble metal catalyst such as platinum has a high capacity for catalytic reaction.

[0022] The exhaust gas purifying filter is preferably made by sintering at least one kind of material selected from among cordierite, silicon carbide and zirconium phosphate powder. This provides an exhaust gas purifying filter that has the pores of the characteristics described above.

[0023] The inner walls of the pores of the porous walls are preferably coated with alumina and the catalyst is supported thereon. Most alumina materials have large specific surface areas in a range from 100 to 200 g per liter. Accordingly, a larger area is available for the catalytic reaction that oxidizes the particulate matter, thereby accelerating the process of oxidizing and removing the particulate matter.

[0024] The exhaust gas purifying filter has honeycomb structure and is made in a cylinder having circular or oval cross section.

[0025] The exhaust gas purifying filter are mostly made in dimensions of 0.8 to 2.5 mm on one side of each cell, 0.1 to 0.6 mm in thickness of the porous wall, 50 to 300 mm in diameter and 50 to 200 mm in length, although the dimensions are not restricted to these values.

[0026] The porosity and mean pore size of the porous wall and the value of ratio Y/X in the second aspect of the invention are similar to these of the first aspect of the invention

[0027] According to the second aspect of the invention, the mixture preferably includes carbon added thereto for the

an advantage of energy saving effect.

[0047] The cordierite is preferably formed by firing a mixture of talc, fused silica and aluminum hydroxide, with the mean particle size of aluminum hydroxide being set in a range from 5 to 20 μm, while total weight A of all particles included in the mixture and the cumulative weight B of particles included in the mixture having sizes of 100 μm and larger satisfy the relation of inequality $B/A \leqq 0.05$.

[0048] When the mean particle size of aluminum hydroxide is in the range described above, pores of the exhaust gas purifying filter can be controlled so that the mean pore size falls within a range from 30 to 50 μm during manufacturing. Also, because the total weight A of all particles included in the mixture and the cumulative weight of particles included in the mixture having sizes of 100 μm and larger satisfy the relation of inequality described above, the particulate matter can be captured with a high probability.

[0049] When the mean particle size of aluminum hydroxide is smaller than 5 μm, mean pore sizes may become too small to keep the pressure loss within a reasonable level. When the mean particle size of aluminum hydroxide is larger than 20 μm, significant number of pores become larger than 100 μm thus making it easy for the particulate to pass through the exhaust gas purifying filter.

[0050] If $B/A > 0.05$, a larger proportion of the particulate matter may pass through the filter.

[Examples]

[0051] Embodiments of the present invention will now be described in detail below.

[0052] In this example, ten kinds of exhaust gas purifying filter having different chemical compositions and different pore characteristics were manufactured. Among these, samples 6 through 9 are the exhaust gas purifying filters according to the present invention, and samples 1 through 5 and sample 10 are reference samples. All the samples were based on cordierite as the main component with a chemical composition consisting of 45 to 55% by weight of SiO_2, 33 to 42% by weight of Al_2O_3, and 12 to 18% by weight of MgO.

[0053] The exhaust gas purifying filter 7 has a cylindrical honeycomb structure of a circular cross section including a multitude of cells 1 that extend along the longitudinal direction as shown in Fig. 1 and Fig. 2. Half the cells serve as introduction passages 2 and the rest serve as exhaust passages 3. The cells are disposed alternately in the vertical and horizontal directions in the cross section in a checkerwork arrangement, while being separated from each other by porous walls 5. The surfaces of the porous walls 5 and the inner surfaces of pores 50 hold catalyst layers 51 having thickness of several micrometers and comprising alumina and a noble metal catalyst supported thereon.

[0054] The introduction passages 2 are open on the exhaust gas introducing side, and are closed with plugs 42 on the other end. The exhaust passages 3 are closed with plugs 43 on the exhaust gas introducing side and are open on the other end. Density of the cells 1 in the cross section perpendicular to the longitudinal direction of the exhaust gas purifying filter is 300 per 6.45 cm^2. The cell 1 measures 1.17 mm along one side of the cross section, and thickness of the porous wall 5 is 0.3 mm. The exhaust gas purifying filter measures 103 mm in diameter and 155 mm in length.

[0055] To capture the particulate matter with the exhaust gas purifying filter 7, the exhaust gas that includes the particulate is introduced into the introduction passages 2 as shown in Fig. 2. The exhaust gas that is introduced then passes through the porous walls 5 and enters the exhaust passages 3, since the introduction passages 2 are closed at the exhaust side.

[0056] At this time, the particulate matter 59 is captured in the pores 50 of the porous walls 5, as shown in Fig. 3. The exhaust gas is discharged from the open ends of the exhaust passages 3. Part of the particulate matter 59 may pass through the pores 50 (arrow A). If the pores 50 are larger than 100 μm, in particular, the particulate matter 59 often passes through the pores 50.

[0057] The particulate matter captured by the porous walls 5 reacts with oxygen included in the exhaust gas under the high temperature condition of the exhaust gas, so as to be oxidized and removed. The reaction is accelerated by the catalyst supported on the porous walls, so that oxidization proceeds efficiently.

[0058] To manufacture the exhaust gas purifying filter, cordierite forming material of the kind and quantity shown in Table 1 and Table 2 and combustible organic material were prepared.

[0059] Table 1 shows mixing proportions of the cordierite forming material and the pore forming material, and Table 2 shows the mean particle size of the cordierite forming material with the ratio of large particles of the cordierite forming material also being shown.

[0060] The ratio of large particles refers to the ratio B/A of total weight A of the cordierite forming material to the cumulative weight B of particles included in the cordierite forming material having sizes of 100 μm and larger.

[0061] As the cordierite forming material, talc, fused silica and aluminum hydroxide are used. As the pore forming material, unexpanded material, expanded material and/or carbon are used. The unexpanded material consists of encapsulized hydrocarbons that expand 40 times in volume at a temperature from 80 to 100°C. For the expanded material, expandable beads that had been expanded with the unexpanded material added beforehand are used.

[0062] Predetermined quantities of an organic binder and water were added to a basic stock material consisting of

(Table 1) Mixing proportion

(% by weight)

Samples	Cordierite forming material			Pore forming material		
	Talc	Fused silica	Aluminum hydroxide	Unexpanded material	Expanded material	Carbon
1	38	18	44	2	-	20
2	38	18	44	2	-	20
3	38	18	44	2	-	10
4	38	18	44	-	-	10
5	38	18	44	2	-	-
6	38	18	44	5	-	20
7	38	18	44	-	5	20
8	38	18	44	-	5	20
9	38	18	44	5	5	20
10	38	18	44	10	20	-

(Table 3)

Samples	Porosity (%)	Mean particle size (μm)	Pressure loss (KPa)	Capturing efficiency (%)	Condition of capturing	Strength (MPa)
1	50	25	0.54	95	accumulated only on the surface	2.1
2	52	27	0.52	95	accumulated only on the surface	1.6
3	48	30	0.59	93	infiltrated the inside	1.9
4	46	32	0.61	94	infiltrated the inside	3.1
5	38	43	0.76	90	infiltrated the inside	3.5
6	66	46	0.42	88	infiltrated the inside	1.2
7	72	48	0.40	88	infiltrated the inside	0.9
8	74	50	0.38	89	infiltrated the inside	1.0
9	78	50	0.35	87	infiltrated the inside	0.8
10	84	72	0.31	55	infiltrated the inside	0.3

4. The exhaust gas purifying filter according to claim 1, wherein inner surfaces of the pores of said porous walls are coated with alumina whereon the catalyst is supported.

5. A method of manufacturing an exhaust gas purifying filter, made of a ceramic material in a honeycomb structure, comprising introduction passages for introducing exhaust gas that includes particulate matter emitted from an internal combustion engine, porous walls that collect said particulate matter and exhaust passages for exhausting the exhaust gas after the particulate matter has been removed therefrom, with said porous walls supporting a catalyst for oxidizing and removing said particulate matter,

the porosity of the porous walls being in a range from 55 to 80%, and mean pore size being in a range from 30 to 50 μm,

while the total volume X of the pores included in said exhaust gas purifying filter and the volume Y of the pores that are not smaller than 100 μm satisfy the relation of inequality $Y/X \leqq 0.05$,

wherein said exhaust gas purifying filter is made by firing a preform that is formed in honeycomb structure from a mixture of ceramic powder and a foaming material so that the foaming material expands during the firing process, and adding the catalyst to be supported thereon.

6. The method of manufacturing the exhaust gas purifying filter according to claim 5, wherein said mixture includes carbon added thereto for the purpose of forming the pores.

7. The method of manufacturing the exhaust gas purifying filter according to claim 5, wherein said foaming material is a mixture of a material of which 5 to 80% has been already expanded and an unexpanded material that expands at a temperature not higher than 100°C.

8. The method of manufacturing the exhaust gas purifying filter according to claim 5, wherein at least one kind of material selected from among cordierite, silicon carbide and zirconium phosphate powder is used as said ceramic powder.

9. The method of manufacturing the exhaust gas purifying filter according to claim 8, wherein said cordierite is formed by firing a mixture of talc, fused silica and aluminum hydroxide.

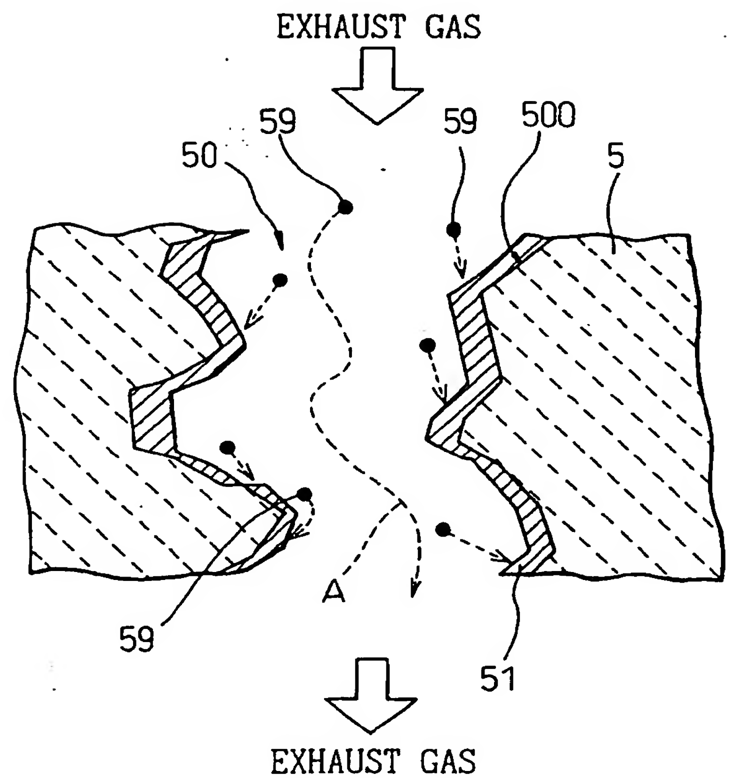
10. The method of manufacturing the exhaust gas purifying filter according to claim 9, wherein the mean particle size of said talc is in a range from 30 to 200 μm, the mean particle size of said fused silica is in a range from 30 to 200 μm and the mean particle size of said aluminum hydroxide is in a range from 5 to 20 μm.

11. The method of manufacturing the exhaust gas purifying filter according to claim 8, wherein said cordierite is formed by firing a mixture of talc, fused silica and aluminum hydroxide,

the mean particle size of said aluminum hydroxide is set in a range from 5 to 20 μm, and

the total weight A of all particles included in the mixture and the cumulative weight B of particles included in the mixture having sizes of 100 μm and larger satisfy the relation of inequality $B/A \leqq 0.05$.

Fig. 3



ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 02 00 7022

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11-07-2002

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